

PATENT ABSTRACTS OF JAPAN

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(54) MANUFACTURE OF TI-AL ALLOY SINTERED BODY

(57)Abstract:

PURPOSE: To manufacture a Ti-Al alloy sintered body excellent in high temp. strength and toughness by kneading Ti powder and Al powder applied with Ni plating having specified thickness with an organic binder, executing compacting and dewaxing, thereafter sintering it in an Ar atmosphere.

CONSTITUTION: The powdery material of Ti powder with Al powder applied with Ni plating having 0.05 to 1.0 μ m thickness is kneaded with an organic binder. As for the Ti powder and Al powder, the one having about $\leq 30\mu$ m average particle diameter is preferably used.

Furthermore, the Ni plating can be executed by a electroless plating. As for the organic binder, the well-known one can be used, and as for the kneading, it is executed preferably by a kneader having high kneading power such as a pressure kneader. Then, the kneaded product is subjected to dewaxing in heating, and after the dewaxing treatment, sintering treatment is executed at 1200°C for about 2hr in the Ar atmosphere. In such a manner, the objective Ti-Al alloy sintered body excellent in high temp. strength and toughness is obtd. and by this method, the manufacture of lightweight heat resistant material parts having a complicated shape is enabled.

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TITLE: Titanium@-aluminium@ alloy sintered compact mfr. for aircraft engines - by mixing **nickel@-plated titanium@ powder** and aluminium@ powder, kneading with organic binder, compacting, degreasing and sintering

PATENT-ASSIGNEE: KAWASAKI STEEL CORP[KAWI]

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ABSTRACTED-PUB-NO: JP 05263165A

BASIC-ABSTRACT:

Ti-Al Alloy sintered compact is made by mixing **Ni-plated Ti-powder** and Al-powder, kneading with an organic binder, compacting the kneaded matter, degreasing and sintering.

USE - Used for aircraft engines. It has excellent high-temp. strength and toughness.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS: TITANIUM@ ALUMINIUM@ ALLOY SINTER COMPACT MANUFACTURE
AIRCRAFT
ENGINE MIX NICKEL@ PLATE TITANIUM@ POWDER ALUMINIUM@ POWDER
KNEAD
ORGANIC BIND COMPACT DEGREASE SINTER

DERWENT-CLASS: M26

CPI-CODES: M22-H03B; M26-A02; M26-B06; M26-B06A;

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CLAIMS

[Claim(s)]

[Claim 1] 0.05 micrometers in thickness It is 1.0 above. mum The manufacture approach of the Ti-aluminum system alloy sintered compact characterized by performing shaping processing, cleaning processing, and sintering processing in Ar ambient atmosphere after kneading Ti powder which performed the following nickel plating, and the mixed powder of aluminum powder with an organic binder.

[Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]**[0001]**

[Industrial Application] This invention relates to the manufacture approach of a Ti-aluminum alloy sintered compact that use is expected as a lightweight heat-resisting material.

[0002]

[Description of the Prior Art] Efficient-izing and lightweight-ization are mentioned as a property required of a jet engine, an internal combustion engine, etc. Among these, about efficient-izing, it is required to raise an engine operating temperature. On the other hand, lightweight-ization cannot be attained although the current nickel radical heat-resistant alloy etc. is used. On the other hand, although the ceramics has come to be used for car motor from a viewpoint of lightweight-izing, dependability poses a problem from the point inferior to toughness. As an ingredient which solves these troubles, the intermetallic compound of a Ti-aluminum system has attracted attention in recent years. It is thought that a field of the invention will expand it from now on since this ingredient is lightweight, and excellent in high temperature strength and rich in toughness from the ceramics.

[0003] The manufacturing method of the sintered compact used as components poses a problem here. After making it the plate like a ferrous material, it was not able to be said that fabrication was carried out to the last configuration. Therefore, the so-called near net shape processing is needed. In this case, powder-metallurgy processing can be considered to be precision casting as the shaping approach (for example, "the reinforcement of an intermetallic compound, deformation", the Japan Institute of Metals symposium draft, 1988, P20).

[0004]

[Problem(s) to be Solved by the Invention] However, when using precision casting, since activity [Ti], a device is needed for an ambient atmosphere, refractories, etc. On the other hand, in general powder-metallurgy processing, manufacture of a complicated geometry component and large-sized components is difficult. Moreover, when performing a powder raw material by dissolution-ingot-grinding, a problem remains in the dissolution and an ingot manufacturing method as well as casting.

[0005] In recent years, the injection-molding method has attracted attention as the manufacture approach of the components of a complicated configuration metal or the ceramics. This is the approach of manufacturing a sintered compact through cleaning and a sintering process, after fabricating the raw material manufactured by carrying out heating kneading of a metal or ceramic fines, and the organic binder by the injection-molding method. However, utilization is not carried out in order to be anxious about penetration of C from an organic binder component, and degradation of the toughness by oxidation at cleaning and a sintering process about active metals, such as Ti. moreover -- although there is a report (a 1st super-environment-resistant advanced ingredient symposium draft (1990.10 months), P7) of injection molding which used Ti-aluminum alloy powder about injection molding of Ti-aluminum system alloy powder, even if the sintered compact of high density is obtained -- a front face -- there is a problem in description, dimensional accuracy, etc. Moreover, as the manufacture approach of Ti sintered compact by injection molding, the reaction of C and O is used for JP,2-54733,A by vacuum

sintering, and the method of reducing C and O is indicated. However, it is necessary to adjust C of a Plastic solid, and the amount of O by this approach. This invention aims at offering the manufacture approach of the Ti-aluminum alloy sintered compact which solved the above troubles.

[0006]

[Means for Solving the Problem] This invention is 0.05 micrometers in thickness. It is 1.0 above. μm After kneading Ti powder which performed the following nickel plating, and the mixed powder of aluminum powder with an organic binder, it is the manufacture approach of the Ti-aluminum system alloy sintered compact characterized by performing shaping processing, cleaning processing, and sintering processing in Ar ambient atmosphere.

[0007]

[Function] According to this invention, nickel which covered the powder front face prevents penetration of C in a kneading process, a forming cycle, and a cleaning process. The complicated configuration sintered compact of high density is obtained without degrading the quality of the material by this. The thickness of nickel plating is 0.05 micrometers. When it is the following, there is no above-mentioned effectiveness. Moreover, 1 μm If it exceeds, the above-mentioned effectiveness will be saturated and will degrade the quality of the material of sintering material. On the other hand, although especially the manufacture approach of Ti powder or aluminum powder that is a raw material is not specified, mean particle diameter is 30 micrometers. The following is desirable (refer to JP,2-54733,A). Since decomposition temperature etc. is not especially restricted, binding material may also use a well-known cementitious material. As the kneading approach, the high kneading machine of kneading force, such as a pressurized kneader and a continuous extrusion machine, is suitable. As the shaping approach, injection molding is effective about a small complicated geometry component. Moreover, when tabular components are required, extrusion molding and roll forming are suitable. At the time of sintering, since nickel reacts with Ti and reduces the melting point, it can make sintering temperature low.

[0008]

[Example] the inside of the electroless deposition liquid (PH5, 90 degrees C) which consists Ti powder with a mean particle diameter of 25 micrometers of chlorination nickel 30g/1, sodium hypophosphite 10g/1, and sodium citrates 10g/1 -- supplying -- predetermined time amount stirring -- it carried out. The acetone washed the powder after plating. In addition, the plating nickel component was 10% in weight %. This powder and aluminum powder with a mean particle diameter of 10 micrometers were kneaded with the pressurization mold kneader with thermoplastics, the wax, and the plasticizer after mixing by 65:35 at the weight ratio, and the injection-molding raw material was produced. After performing heating cleaning by carrying out injection molding of this to tabular [with die length of 80mm, a width of face / of 15mm /, and a thickness of 4mm], and carrying out a temperature up with the heating rate of 10 degrees C/h to 550 degrees C in nitrogen gas, sintering of 2 hours was performed at 1200 degrees C in Ar ambient atmosphere.

[0009] It was with Ti powder which does not perform nickel plating as comparison material, and the sintered compact was manufactured at this process (example 1 of a comparison). Moreover, the sintered compact was manufactured at this process using the powder which mixed the carbonyl nickel powder equivalent to the amount of plating with Ti powder and aluminum powder (example 2 of a comparison). Moreover, the case (example 3 of a comparison) where average plating thickness is 0.04 micrometers, and 1.2 micrometers (example 4 of a comparison) nickel-plating Ti powder were manufactured, and the sintered compact was manufactured at this process. From these sintering material, the test piece was processed and the mechanical property was investigated. Both quality-of-the-material property is shown as compared with Table 1. It turns out that the sintered compact by this invention is excellent in the reinforcement in an elevated temperature, and ductility compared with the example of a comparison.

[0010]

[Table 1]

	メッキ 厚さ (μm)	相対焼結 密度 (%)	TS (室温) (kgf/mm^2)	E1 (室温) (%)	0.2%耐力 (800℃) (kgf/mm^2)
実施例1	0.1	95	58	1.8	40
実施例2	0.5	96	62	1.5	48
実施例3	0.8	97	64	1.2	52
比較例1	0	90	40	0.2	30
比較例2	0	90	60	0.1	45
比較例3	0.04	90	48	0.2	40
比較例4	1.2	90	65	0.6	42

[0011]

[Effect of the Invention] While manufacture of the lightweight heat-resisting-material components of a complicated configuration is attained by this invention and being able to measure engine high performance-ization, it leads to application expansion of an intermetallic compound.

[Translation done.]

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(54)【発明の名称】 Ti-Al系合金焼結体の製造方法

(57)【要約】

【目的】航空機のエンジンなどに用いるため、高温強度と靱性にすぐれたTi-Al系金属間化合物による複雑形状の軽量耐熱材料部品の製造を可能とする。

【構成】金属粉末射出成形において、Niメッキを施したTi粉末とAl粉末とを混合後、有機バインダーと混練し、成形処理、脱脂処理、焼結処理を行なうことにより、Ti-Al系合金焼結体を得る。

【特許請求の範囲】

【請求項1】 厚さ0.05 μm 以上1.0 μm 以下のNiメッキを施したTi粉末と、Al粉末の混合粉末とを有機バインダーと混練したのち、成形処理、脱脂処理、Ar雰囲気中での焼結処理を行なうことを特徴とするTi-Al系合金焼結体の製造方法。

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明は、軽量耐熱材料として利用が期待されているTi-Al合金焼結体の製造方法に関する。

【0002】

【従来の技術】ジェットエンジン、内燃機関等に要求される特性として高効率化、軽量化が挙げられる。このうち高効率化についてはエンジンの運転温度を高めることが必要である。これに対し、現在Ni基耐熱合金等が使用されているが、軽量化は達成できない。一方軽量化という観点から、自動車用エンジンにはセラミックスが使用されるようになってきたが、靱性に劣る点から信頼性が問題となる。これらの問題点を解決する材料として、近年Ti-Al系の金属間化合物が注目されてきた。この材料は軽量でかつ高温強度に優れ、セラミックスよりも靱性に富むため、今後利用分野が拡大していくと考えられる。

【0003】ここで問題となるのが、部品となる焼結体の製造法である。鉄鋼材料のように板にしてから最終形状に成形加工するといったことは出来ない。従って、いわゆるニアネットシェイプ加工が必要となってくる。この場合、成形方法として精密鋳造と粉末冶金法が考えられる（例えば「金属間化合物の強度と変形」、日本金属学会シンポジウム予稿、1988、P20）。

【0004】

【発明が解決しようとする課題】しかしながら、精密鋳造法を利用する場合、Tiが活性なため雰囲気、耐火物等に工夫が必要となる。一方、一般の粉末冶金法では複雑形状部品、大型部品の製造がむずかしい。また粉末原料を溶解-インゴット-粉砕で行なう場合、鋳造法と同じく溶解およびインゴット製造法に問題が残る。

【0005】近年、複雑形状金属やセラミックスの部品の製造方法として、射出成形法が注目されてきている。これは金属あるいはセラミックス微粉と有機バインダを加熱混練することにより製造した原料を射出成形法にて成形した後脱脂、焼結工程を経て焼結体を製造する方法である。しかしながらTi等の活性金属については脱脂、焼結工程で有機バインダ成分からのCの進入や、酸化による靱性の劣化が懸念されるため実用化はされていない。また、Ti-Al合金粉末の射出成形については、Ti-Al合金粉末を用いた射出成形の報告（第1回超環境性先進材料シンポジウム予稿（1990・10月）、P7）があるが、高密度の焼結体は得られても

表面性状、寸法精度などに問題がある。また、射出成形によるTi焼結体の製造方法としては、特開平2-54733号公報に真空焼結によりCとOの反応を用いて、C、Oを低減させる方法が開示されている。しかし、この方法では成形体のC、Oの量を調整する必要がある。本発明は、以上のような問題点を解決したTi-Al合金焼結体の製造方法を提供することを目的とする。

【0006】

【課題を解決するための手段】本発明は、厚さ0.05 μm 以上1.0 μm 以下のNiメッキを施したTi粉末と、Al粉末の混合粉末とを有機バインダーと混練したのち、成形処理、脱脂処理、Ar雰囲気中での焼結処理を行なうことを特徴とするTi-Al系合金焼結体の製造方法である。

【0007】

【作用】本発明によれば、粉末表面を覆ったNiが混練工程、成形工程、脱脂工程でのCの進入を防ぐ。これにより材質を劣化させることなく、高密度の複雑形状焼結体が得られる。Niメッキの厚さが0.05 μm 未満の場合、上記効果がない。また1 μm を超えると、上記効果は飽和し、焼結材の材質を劣化させる。一方、原料であるTi粉末やAl粉末の製造方法は特に規定しないが、平均粒径は30 μm 以下が望ましい（特開平2-54733号参照）。結合材も特に分解温度等は制限されないため、公知のバインダー材を使用してよい。混練方法としては、加圧ニーダ、連続押出機等の混練力の高い混練機が適する。成形方法としては、小型複雑形状部品については、射出成形が有効である。また、板状の部品が必要な場合は押し出し成形、ロール成形が適する。焼結時NiはTiと反応して融点を低下させるため、焼結温度を低くすることができる。

【0008】

【実施例】平均粒径25 μm のTi粉末を塩化Ni30g/1、次亜リン酸ナトリウム10g/1、クエン酸ナトリウム10g/1からなる無電解メッキ液（PH5、90℃）中に投入し、所定の時間攪拌した。メッキ後の粉末をアセトンで洗浄した。なお、メッキNi成分は重量%にて10%であった。この粉末と平均粒径10 μm のAl粉末を重量比にて65：35で混合後、熱可塑性樹脂、ワックス、可塑剤とともに加圧型ニーダにて混練し、射出成形原料を作製した。これを長さ80mm、幅15mm、厚さ4mmの板状に射出成形し、窒素ガス中で550℃まで10℃/hの加熱速度で昇温することにより加熱脱脂を行なった後、Ar雰囲気中で1200℃で2時間の焼結をおこなった。

【0009】比較材としてNiメッキを施さないTi粉末をもちいて同工程で焼結体を製造した（比較例1）。また、メッキ量に相当するカルボニルNi粉末を、Ti粉末、Al粉末と混合した粉末を用いて同工程にて焼結体を製造した（比較例2）。また、平均メッキ厚さが

0.04 μ mの場合(比較例3)と1.2 μ m(比較例4)のニッケルメッキTi粉末を製造し同工程にて焼結体を製造した。これらの焼結材より試験片を加工し、機械的特性を調査した。両者の材質特性を表1に比較して*

*示す。本発明による焼結体は比較例に比べて高温での強度、延性に優れることがわかる。

【0010】

【表1】

	メッキ 厚さ (μ m)	相対焼結 密度 (%)	TS (室温) (kgf/mm ²)	E1 (室温) (%)	0.2%耐力 (800℃) (kgf/mm ²)
実施例1	0.1	95	58	1.8	40
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比較例3	0.04	90	48	0.2	40
比較例4	1.2	90	65	0.6	42

【0011】

【発明の効果】本発明により複雑形状の軽量耐熱材料部※

※品の製造が可能となり、エンジン等の高性能化が計れるとともに、金属間化合物の用途拡大につながる。